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SAXS for In-situ Structure-Reactivity Studies of Nanostructured Aluminum Trifluoride

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Beamline(s): X10A

Introduction: Plasma decomposition of zeolite gives rise to nanostructured AlF_3, showing agglomerations of 4-5 nm particulates by high-resolution TEM. Small-angle X-ray scattering (SAXS) was used to monitor the evolution of these features with temperature. Simultaneous temperature-programmed reaction experiments with CHF_2Cl were also performed to study possible structure-reactivity correlations.

Methods and Materials: Small-angle X-ray scattering technique was performed in all experiments using a 2D-CCD detector. The nanostructured, amorphous AlF_3 materials were prepared by plasma decomposition of zeolite described elsewhere.[1] Fe- and K-incorporated AlF_3 was obtained by similar procedures. A special flow cell apparatus (Dr. John Hansen and Peter Chupas, SUNY-Stony Brook) allowed temperature-dependent structural changes to be measured in-situ during temperature-programmed reaction with CHF_2Cl. The air-sensitive material was packed in an N_2-filled glove box before mounting.

Results: No small-angle X-ray scattering was observed in any sample. Increasing the temperature identified phase transformations in the AlF_3 system, but did not reveal any SAXS information. The absence of small-angle X-ray scattering in our materials may have originated from poorly defined homogeneity in particulate size throughout the majority of our sample and/or densification over the time of synthesis to analysis.

Conclusions: In-situ SAXS during temperature-programmed reaction experiments with CHF_2Cl was studied for zeolite-derived AlF_3. It was expected that changes in SAXS might lead to structural-reactivity correlations with temperature-programmed reaction data. Unfortunately, no small-angle X-ray scattering was present at any temperature for any of our materials. Reasons for the absence of SAXS in our materials are currently being considered.

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References: J.L. Delattre, P.J. Chupas, C.P. Grey, A.M. Stacy, “Plasma-Fluorination Synthesis of High Surface Area Aluminum Trifluoride from a Zeolite Precursor,” Journal of the American Chemical Society, **123**, 5364-5365.